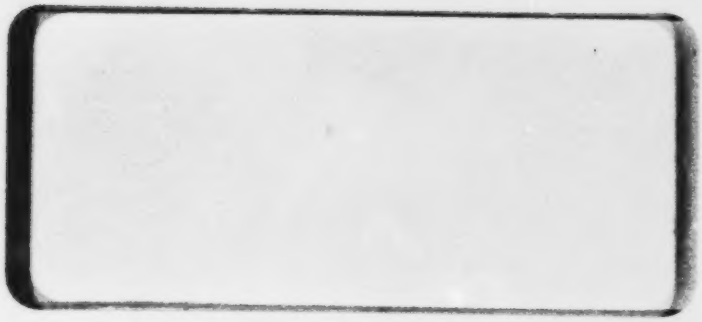
APPLIED RESEARCH REPORT



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A STUDY ON THE DESULPHURIZATION OF COAL BY BACTERIAL LEACHING

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MICROMEDIA

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SUMMARY

1. A pilot bacterial leaching experiment was conducted on Prince Mine coal (Hub seam) for two particle sizes.
   Phase I run-of-mine 1" - 4"
   -leached for 12 months (completed).

   Phase II run-of-mine 1/2" - 1/4"
   -leached for 4 months (continuing)

2. Total sulphur content of the coal was reduced 20% with pyritic sulphur reduced 40% during leaching.

3. Coal innoculated with Thiobacillus ferroxidans did not show a significant difference in the rate of pyrite oxidation compared to coal without innoculation. This is likely due to the presence of indigenous bacteria in the control pile.

4. At least six different hydrated sulphate minerals have precipitated onto the coal. A complex paragenesis is indicated which may relate to the relative influence of chemical versus microbial oxidation activity.

5. The interpretation of data from this leaching experiment is hampered by two factors:

   a) non-representative sampling of a coal pile.

   b) lack of sterility in the control pile and thus the influence of indigenous bacteria.

INTRODUCTION

One of the significant problems related to the utilization of Eastern Canadian Coal is the sulphur content. Research conducted by Dr. R.G.L. McCready at Dalhousie University has indicated that microbial action can significantly reduce the sulphur content of Minto Coals (McCready, 1985). The present study was conducted at the Atlantic Coal Institute as a follow-up to the research initiated by Dr. McCready. A pilot leaching experiment was set up to determine the applicability of microbial desulphurization to Sydney Basin bituminous coal. The study involved investigation of the leaching of sulphur from run-of-mine coal by the action of Thiobacillus ferrooxidans in a pilot scale setting using 200 kg batches of coal in bins. Phase I of this study formed DSS Serial #OSC84-00113, File #12SC.31028-4-4675 and was carried out at the Atlantic Coal Institute during 1984-85. The present report forms Phase II as a revised leaching experiment and new analytical data on both Phase I and II collected during 1985-1986.

The present study, Phase II, involves further analysis of the materials in Phase I and a continuation of the leaching experiment with modified conditions. In particular, this study addressed the nature of the hydrated sulphates formed by the microbial action or oxidation of pyrite in Phase I. Scanning electron microscopy (SEM) and Energy Dispersive X-Ray (EDX) analysis were applied to characterize the morphology and chemistry of these mineral species. The coalpiles from Phase I were screened to determine

structural breakdown and analysed for ash, sulphur forms and calorific value to monitor changes. Samples of the final leachate were collected and sent for trace element analysis to determine levels of potential pollutants.

For investigation of the degree of sulphur removal, two pilot leaching systems were constructed at the laboratories of the Atlantic Coal Institute in Sydney, Nova Scotia. One system employed a 200 kg batch of coal from Prince Mine. The coal was screened to +1/4" - -1/2" to allow for more representative sampling as compared to the coal used in Phase I sized at +1" - 4". This coal pile was rinsed intermittently with a solution innoculated with T. ferrooxidans and acidified to inhibit growth of ciliate or zooflagellate predators of the bacteria. A second leaching system, consisting of the same materials, was set up as a control without bacterial innoculation and chlorine was added to inhibit indigenous bacteria. The level of ferric iron (Fe^{3+}) in solution was monitored as an indirect indication of the level of pyrite removal. An alternative method would have been to monitor sulphate levels (Taylor et al 1984).

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PROJECT MILESTONES

1. Collect representative hydrated sulphates from the control and innoculated pile of Phase I and investigate morphology and chemistry by SEM-EDX.
2. Conduct sieve analysis, ash, S, sulphur forms, BTU on innoculated and control coal pile from Phase I study.
3. Modify pilot leaching experiment for particle size, control pile isolation and sterilization. Analyse starting materials.
4. Continue intermittent spraying of coal piles using distilled water, bacteriocide solution and Thiobacillus ferrooxidans solution as appropriate. Collect representative samples of leached coal and effluent at appropriate intervals and analyse by INAA, ASTM methods and spectroscopy as appropriate. Leaching should continue for 8 months.
5. Data compilation and report preparation.

TECHNICAL APPROACH

Hydrated Sulphates

Specimens representing the oxidation products (yellow and white material - see photographs Appendix 4) on the surface of the coal were collected from the coalpiles of both the innoculated and non-innoculated leaching tanks from Phase I. Representative fragments of these materials were mounted on carbon SEM stubs using carbon paint and carbon coated using a Polaron ES5100 Sputter coater. These samples were then analysed with a JEOL JSM-T300 Scanning Electron microscope to provide morphological information on these species. X-ray spectra were obtained using a Tracor-Northern TN5500 Energy dispersive x-ray analyser to show the elemental composition of the samples.

Phase I: Final Tests

At the end of the leaching period of 12 months, the coalpiles from both Phase I tanks were removed and sieved through a 1" mesh screen to determine structural breakdown. The piles were recombined and splits were taken for sulphur, sulphur forms, ash, moisture and calorific value (ASTM procedures D2492 and D3174) determinations.

Phase II: Revised Pilot Study

Two batch scale leaching apparati were set up as in figure 1. Approximately 400 kg of run-of-mine coal, screened at -1/2" + 1/4" was collected from stockpiles at Prince Mine representing high volatile Bituminous coal from the Hub Seam.

This sample was split, a subsample removed, and the remainder was split into two "equal" piles and placed in the leaching tanks. The subsample was analysed for moisture, ash, sulphur, sulphur forms, and calorific value (ASTM procedures) to provide initial characteristics. The coalpiles were rinsed with water to remove fines and soluble salts prior to initiation of leaching.

Nutrient solutions were prepared as in Appendix 1. One solution (approximately 25 l) was innoculated with a culture of T. ferrooxidans and added to one leaching tank. Another 25 l solution was sterilized with 250 mls chlorine bleach and added to the control tank. Samples were removed and analysed spectrophotometrically for ferric iron (Fe^{3+}) content (Appendix 2) to establish initial Fe^{3+} concentrations.

For the next few months, the coalpiles were rinsed intermittently with the solutions, and the liquid levels were checked and maintained with acidified distilled water. Samples of the

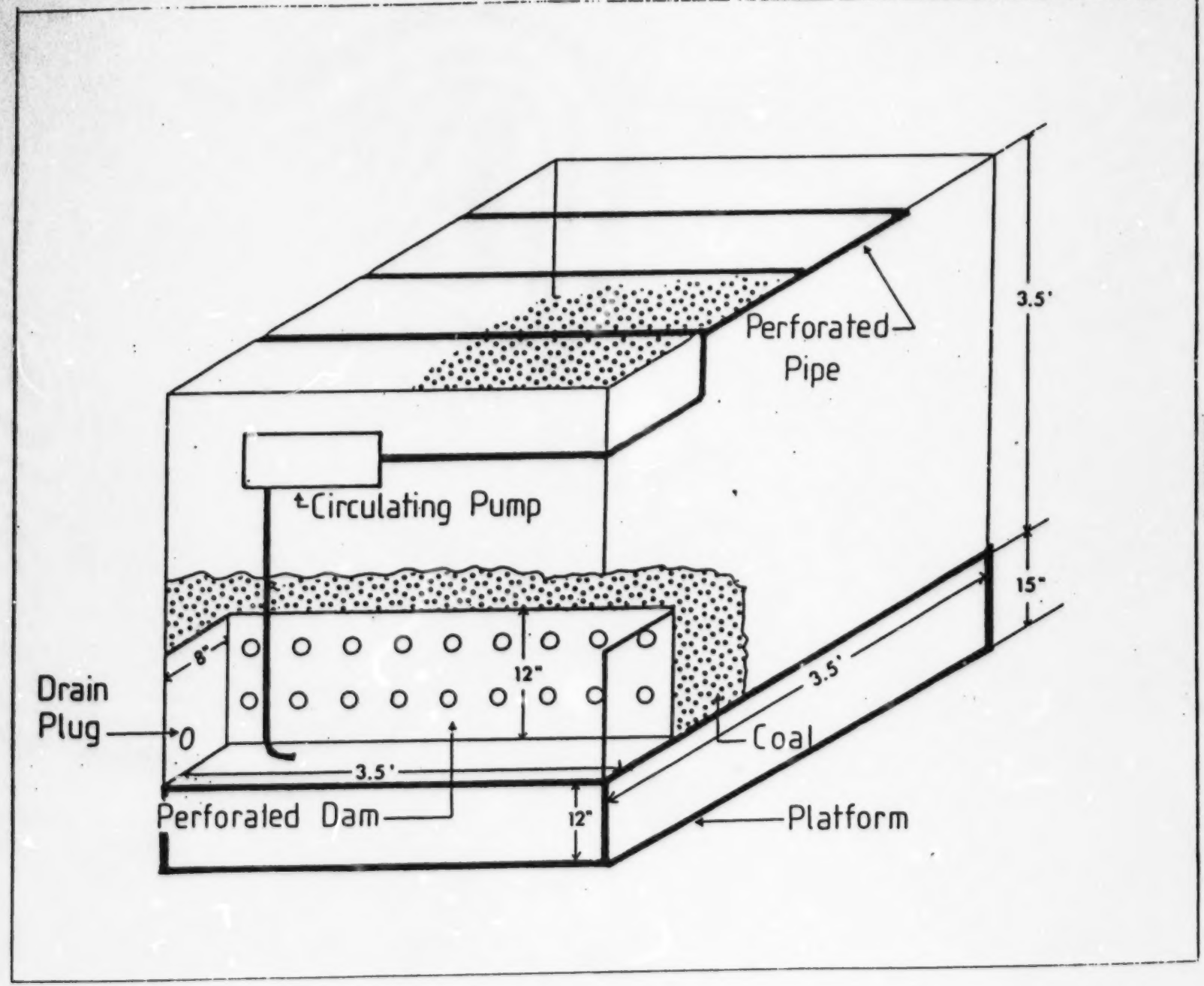

Perforated
Pipe
3.5'
Circulating Pump
15"
8"
12"
3.5'
Drain
Plug
Coal
3.5'
Perforated Dam
12"
Platform

FIGURE 1

leaching solutions were collected weekly for the first three months and analysed for Fe^{3+} content. Samples from the control tank showed significant increases in Fe^{3+} at the end of the first month. The control solution showed a viable culture of T. ferrooxidans (Appendix 1) and more chlorine bleach was added. This did not result in sterilization. Further attempts to maintain bacteriostasis in the control tank were abandoned at the suggestion of the scientific authority.

After an initial three month leaching period, samples of the leachate were collected and sent for neutron activation analysis (INAA) to Neutron Activation Services, (Hamilton,Ontario) and Atomic absorption spectrophotometry (AA) to Dr. M. Zentilli at Dalhousie University. Samples of coal were collected using a cylindrical core sample collection device. These samples were analysed for moisture, ash, sulphur, sulphur forms and calorific value.

The full leaching term was not completed at time of writing. At the end of the eight month leaching period samples will be collected and analysed as above and the coalpiles will be sieved to determine structural integrity of coal particles.

RESULTS

Leaching Results: Phase I

Analysis of the coal from Phase I (Table 1) shows that the total sulphur content of the coal in the tanks was reduced approximately 20%. The pyritic sulphur content showed a 40% reduction while the sulphate sulphur increased by more than 10 fold as would be expected from precipitated hydrated sulphates produced by the oxidation of pyrite. The interim coal samples (Feb. 10/86) show wide ranging and unexpected results (i.e. increases in pyritic sulphur). This variability has been attributed to non-representative sampling of the large top size coalpiles. A proper comparison should, therefore, consider ratios rather than absolute values. The ash content of the samples showed no appreciable changes. This indicates that precipitated sulphates and iron oxides balance approximately the loss in pyrite. Sieve analysis shows that 30% of both coalpiles had been broken down to less than one inch diameter. We conclude that the addition of bacteria has not significantly enhanced weathering.

In Phase I, the ferric iron (Fe^{3+}) concentration in both tanks showed a steady increase up to March 1985 (Figure 2 and Table 1) suggesting removal of pyrite. The apparent drop in Fe^{3+} concentration after this time is partially attributed to solution

Table 1.

MICROBIAL LEACHING - PHASE I

A. COAL - CHEMICAL ANALYSIS

DATE	SAMPLE	SULFUR FORMS (%)				ASH (%)	MOISTURE (%)	COMMENT
		sulfate	pyritic	organic	total			
SEPT.25/84	INITIAL SAMPLE	0.02	3.78	0.71	4.52	14.73		RDM SAMPLE
FEB.10/85	INNOC. SURFACE	0.10	0.80	1.52	2.42	6.47	3.11	
	INNOC. SUB-SURFACE	0.11	2.43	0.88	3.42	14.64	3.22	HAND-PICKED
	NOT INNOC. SURFACE	0.13	4.23	0.92	5.28	14.23	2.65	SPECIMENS
	NOT INNOC. SUB-SURFACE	0.22	5.20	0.76	6.18	26.16	4.57	
NOV.15/85	INNOCULATED	0.31	2.09	1.36	3.75	14.65	2.81	SIEVE-SPLIT
	NOT INNOCULATED	0.31	2.35	0.88	3.54	14.99	2.89	SAMPLES

B. COAL - PHYSICAL ANALYSIS

- Sieve analysis on coalpiles after leaching period of 12 months

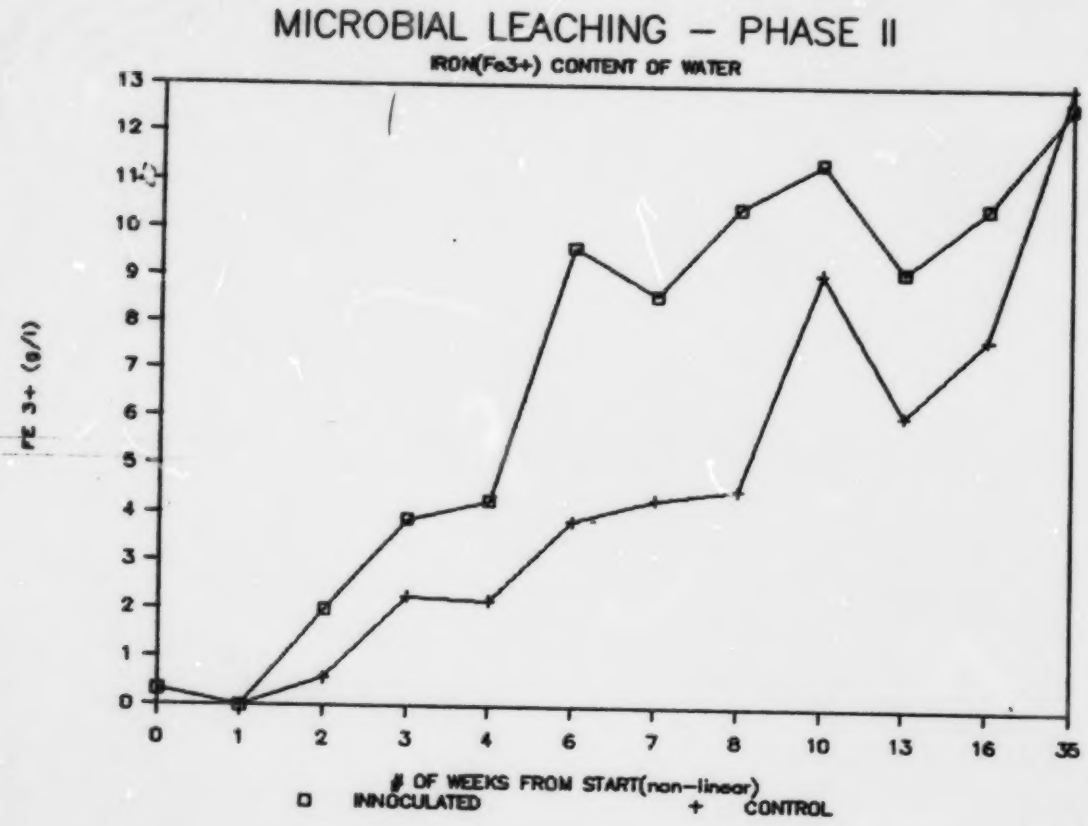
INNOCULATED COALPILE	29% < 1"
NOT INNOCULATED COALPILE	30% < 1"

C. LEACHATE - CHEMICAL ANALYSIS

Fe+3 (mg/ml)

DATE	innoculated tank	not innoculated tank
OCT.22/84	1.3	0.7
NOV.2/84	3.0	2.7
NOV.12/84	4.5	3.1
NOV.23/84	6.7	5.0
DEC.3/84	6.3	6.5
DEC.10/84	10.0	8.5
JAN.31/85	10.0	11.8
MAR.31/85	19.0	14.6
MAY 31/85	9.0	11.0
SEPT.30/85	6.8	6.0

FIGURE 2.


MICROBIAL LEACHING – PHASE II
IRON(Fe3+) CONTENT OF WATER
Fe 3+ (g/l)
13
12
11
10
9
8
7
6
5
4
3
2
1
0
0
1
2
3
4
6
7
8
10
13
16
35
OF WEEKS FROM START(non-linear)
□ INNOCULATED
+ CONTROL

leakage from the tanks and precipitation of iron sulphates and oxides. The absolute values have been affected by the addition of distilled water during the course of the experiment to compensate for evaporation losses.

When we compare the innoculated pile and the control pile (see summary chart), there is no significant difference in behaviour with time. A slightly higher reduction in total sulphur occurred in the control pile but slightly less pyritic sulphur appears to have been removed. These variations are within experimental error of the analytical tests for sieve size, sulphur forms and total sulphur. The discrepency in organic sulphur content, reflected by an <u>increase</u> in organic sulphur in the innoculated pile likely reflects the problems inherent to ASTM procedure D2492. Incomplete digestion of micron size pyrite inclusions in coal leads to overestimation of organic sulphur. Within experimental error we must, therefore, conclude that the rate of pyrite leaching was similar in the innoculated versus control pile over the 12 month duration. The natural oxidation of a wet coal pile with indigenous bacteria has not been enhanced by the addition of bacterial culture over the 12 month term. This does not, however, preclude a rate determining influence at the initial days of leaching.

TABLE 3. **SUMMARY CHART**

	Phase I (25 month test)	**Bacterial**	**Control**
1.	Particle size	29% reduction	30% reduction
2.	Ash content	1% reduction	1.8% reduction
3.	Pyrite ratio (% Pyrite S/Total S)		
	initial	0.84	0.84
	interim (surface)	0.33	0.80
	interim (subsurface)	0.71	0.84
	final	0.56	0.66
	reduction.....	28%	18%
4.	Sulphate ratio (sulphate S/Total S) x 100		
	initial	0.44	0.44
	interim (surface)	4.13	2.46
	interim (subsurface)	3.22	3.56
	final	8.27	8.76
	increase.....	18.8X	19.9X
5.	Total Sulphur	reduced 17%	reduced 22%
	Pyritic Sulphur	reduced 45%	reduced 38%

Leaching Results: Phase II

The coal for Phase II had a similar initial total sulphur content but with higher organic sulphur. This may be an analytical discrepency with ASTM D2492. Ash values were lower in the initial sample as well as interim collections.

The Fe^{3+} concentration monitored for both innoculated and control bins showed a steady increase with the innoculated solutions displaying a more rapid rise (Figure 2b, Table 2). No loss of solution has been experienced to date as was reported in Phase I. After 12 weeks, the control solution was at a concentration level some 80% of the innoculated solution. This suggests that the indigenous bacterial activity requires several weeks to match the leaching activity for innoculated coal piles. To quantify this influence requires more data than is available from this project to date.

Oxidation products: SEM-EDX Results

The sulphates that "grew" on the surface of coal samples from both the control and innoculated pile exhibited a variety of morphology and chemistry. Appendices 3 and 4 illustrate the megascopic and microscopic details of the mineral suites. Three basic morphologies were encountered: fibres, massive granulates and hexagonal plates or "books" of plates with sharp or ragged

edges. The chemical results from EDX suggest major elements as follows in various combinations (Table 4): S, Fe, Si, Al, Mg. Based on peak heights (which reflect both chemistry and topography) we can recognize up to six different sulphate species:

1. Fe > S > Si, Al
2. S > Fe > Si, Mg
3. S > Fe > Si, Mg, Al
4. S > Fe > Si, Al
5. S > Fe > Si
6. S > Al > Fe

The presence of Si may be an instrument artifact.

Table 2.

MICROBIAL LEACHING — PHASE II

A. COAL - CHEMICAL ANALYSIS

DATE	SAMPLE	SULFUR FORMS (%)				ASH	MOISTURE	CALORIFIC VALUE	COMMENT
		sulfate	pyritic	organic	total	(%)	(%)	(BTU/lb)	
NOV.19/86	INITIAL SAMPLE	0.01	3.29	1.24	4.54	9.25	3.94		SIEVE SPLIT
MAR.12/86	INNOCULATED PILE	0.00	2.79	0.72	3.51	7.94	4.49	12792	CORE
	CONTROL PILE	0.20	2.47	0.71	3.37	7.69	4.86	12723	SAMPLES
SEPT.5/86	INNOCULATED PILE	0.03	1.93	1.51	3.47	8.50	4.70	12577	SIEVE SPLIT
	CONTROL PILE	0.07	2.48	1.72	4.27	8.16	4.65	12661	SIEVE SPLIT

C. LEACHATE - CHEMICAL ANALYSIS

Fe+3 (mg/ml)

DATE	innoculated	control
DEC.10/85(INITIAL)	0.3	<.020
DEC.13/85	<.020	<.020
DEC.27/85	2.0	0.6
JAN.6/86	3.9	2.3
JAN.10/86	4.3	2.2
JAN.23/86	9.6	3.9
FEB.3/86	8.6	4.3
FEB.12/86	10.5	4.5
FEB.27/86	11.4	9.1
MAR.17/86	9.2	6.1
APR.11/86	10.3	7.8
MAR.25/86	12.6	13.0

Table 4. HYDRATED SULPHATES
MINERAL CHARACTERISTICS

Sample	Megascopic	SEM morphology	EDX Chemistry (peak order)
	Control Pile		
03	white crust	massive	Fe>S>Si,Al
05	orange-yellow massive	hexagonal "books"	S>Fe>Si,Mg
	Innoculated Pile		
14	white, yellow globular	1. webby fibres	S>Fe>Al,Si
		2. granular	S>Fe>Si,Mg
		3. ragged plates	S>Fe>Al,Si
		4. plates, rosettes	S>Fe>Si
17	orange, yellow massive	1. fibres	S>Al>Fe
		2. "books" with ragged edges	S>Fe
18	orange crust	1. fibres, rods	S>Fe>Si,Al
		2. rosettes with ragged plates	S>Fe>Si

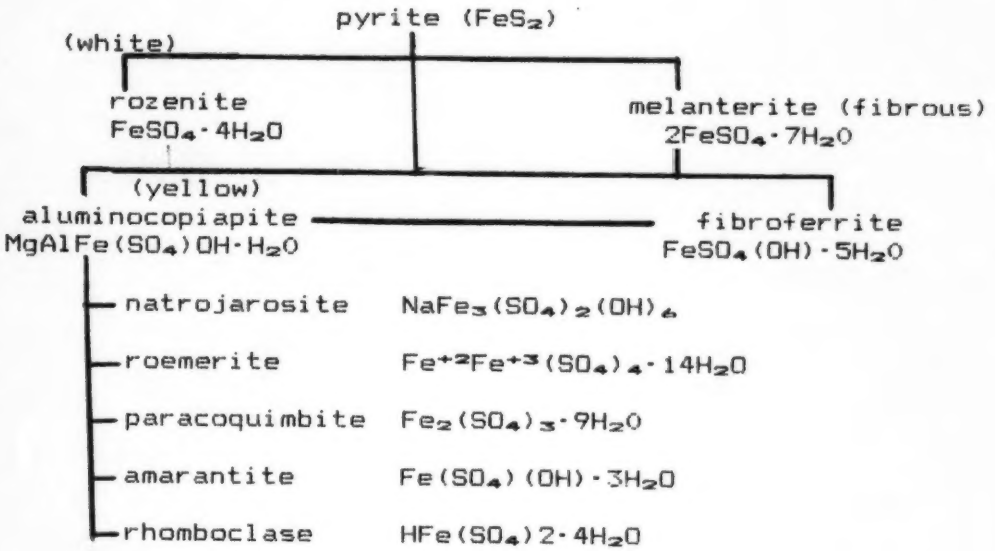
Hydrated Sulphates: Discussion

Zodrow (1980) studied in situ hydrated sulphates from the Prince Mine and concluded that colour was diagnostic of environment. Canary yellow copiapite group minerals crystallized on surface while deep yellow crystals formed underground. Chemical analyses for two copiapite samples from Prince Mine were reported:

Prince Mine Copiapites:	Major Elements							
	Fe	SO_4	Al	Mg	Ca	Na	Mn	K
1. Pyrite alteration:	14.24	48.4	2.46	1.08	0.03	0.16	0.22	0.01
2. Drainage Precipitate:	21.60	47.8	0.10	0.10	0.04	0.14	0.07	0.02

	Trace Elements (ppm)		
	Cu	Ni	Zn
1. Pyrite alteration:	269	400	560
2. Drainage Precipitate	85	128	92

Zodrow (1980) reported that aluminocopiapite samples stored over two years showed a reaction sequence of minerals. Zodrow *et al* (1979) found aluminocopiapite growing on vermicular fibroferrite. Based on their two papers, a cyclic reaction series starting with pyrite has been proposed:



In addition to these series, Zodrow et al (1979) and Zodrow (1980) have reported other hydrated sulphates which "bloom" seasonally at other locations in the Sydney Coalfield including:

pickeringite	$MgAl_2(SO_4)_4 \cdot 22H_2O$
halotrichite	$FeAl_2(SO_4)_4 \cdot 22H_2O$
sideronatrite	$Na_2Fe(SO_4)_2(OH) \cdot 3H_2O$
tamarugite	$NaAl(SO_4)_2 \cdot 6H_2O$
bloedite	$Na_2Mg(SO_4)_2 \cdot 4H_2O$

We have had the opportunity to analyse some of the copiapite group minerals from Zodrow's collection. His data (unpublished) suggests that members of the copiapite group that occur in Sydney coal samples, either in situ or after storage include:

aluminocopiapite
ferrocopiapite
magnesiocopiapite
magnesian ferrocopiapite

Welton (1984) identified "copiapite" as tabular euhedral crystals on the surface of pyrite. Our analysis of some of Zodrow's copiapite group samples suggest that aluminocopiapite occurs as hexagonal blocky crystals while ferrocopiapite shows a more rod-like morphology. This does not correlate with the suite of samples from this project. Our leaching experiment yielded abundant fibrous minerals which tended to be rich in aluminium. The minerals highest in iron tend to have more massive morphology.

It is apparent that the major element chemistry from EDX analysis, and the morphology is not sufficient evidence to assign mineral species names. Nevertheless, we can conclude that a complex mineral paragenesis is evident in the hydrated sulphates recovered from both the control and innoculated tanks. In the absence of XRD supportive data, we speculate that all or some of the sulphate minerals previously mentioned are present. No significant differences in sulphate species can be attributed to bacterial innoculation. The various mineralogy is likely the combined influence of coal mineral distribution (Mg, Al) and aerobic/aquatic environment of growth.

SUMMARY AND RECOMMENDATIONS

The present study has not been sufficient to correlate the significance of hydrated sulphate mineralogy to the microbial action on pyrite oxidation. Nevertheless, the nature of sulphates should have some relationship to the chemical pathways followed by pyrite oxidation.

Under acid conditions (pH <3) two general pathways of pyrite oxidation have been recognized (Taylor et al 1984):

1. By oxygen:

$$2FeS_2 + 7O_2 + 2H_2O \rightarrow 2Fe^{2+} + 4SO_4^{2-} + 4H^+$$

2. By ferric iron:

$$FeS_2 + 14Fe^{+3} + 8H_2O \rightarrow 15Fe^{+2} + 2SO_4^{2-} + 16H^+$$

The second reaction is controlled by availability of ferric iron by the reaction:

3. $4Fe^{+2} + O_2 + 4H^+ \rightarrow 4Fe^{+3} + 2H_2O$

The relative importance of reactions #1 and #2 depend on the availability of oxygen and ferric iron. In the natural environment, such as our pilot study, the oxidation rate is catalysed by Thiobacillus ferrooxidans by either an indirect or direct action. The indirect action involves increasing the availability of ferric iron and thus controlling reaction #2. The direct action results from bacterial attachment to pyrite and direct oxidation of iron and sulphur at the interface.

Taylor et al (1984) conducted laboratory experiments to define the conditions and relative importance of the chemical and microbial pathways in pyrite oxidation. Their experiments for "wet/dry" conditions best correlate to our pilot project and model the fluctuating water levels that would be encountered in coal piles or mines.

The wet/dry innoculated experiments of Taylor et al (1984) indicated that T. Ferrooxidans accelerated pyrite oxidation relative to sterile experiments. Our larger pilot study did not reveal such an acceleration, likely because we could not maintain a sterile control.

Their experiments furthermore revealed that for wet/dry conditions, the oxidation of pyrite proceeded along both chemical and microbial pathways in the proportion of:

1. 77% microbial (reaction 3)
2. 23% chemical (reaction 1)

The relative proportions of chemical to microbial activity under various conditions should lead to differences in ferric iron/ferrous iron levels in solution. This in turn should influence the nature of hydrated sulphate species that precipitate.

We recommend that the relationship of sulphate mineralogy to microbial oxidation of pyrite be further investigated. Under more controlled conditions, the growth of sulphate species may correlate with microbial or chemical activity. Such a study would require more emphasis on mineralogical identification (XRD) as well as sulphur isotopic data to complement the SEM-EDX results.

REFERENCES

1. McCready R.G.L. (1984) Microbiological studies on High Sulphur Coals Part 2. National Research Council, Atlantic Research Laboratory Technical Report 50, NRCC 23601. 75p.

2. Taylor B.E., Wheeler M.C. and Nordstrom D.K. (1984) Stable isotope geochemistry of acid mine drainage: experimental oxidation of pyrite. Geochimica et Cosmochimica Acta Vol. 48, pp. 2669-2678.

3. Welton J.E. (1984) SEM Petrology Atlas. Amer. Association Petrol Geologists, Tulsa Oklahoma, U.S.A., page 212.

4. Zodrow E.L. (1980) Hydrated sulfates from Sydney Coalfield, Cape Breton Island, Nova Scotia, Canada: the copiapite group. American Mineralogist, Vol. 65, p. 961 - 967.

5. Zodrow E.L., Wiltshire J. and McCandlish K. (1979) Hydrated sulfates in the Sydney Coalfield of Cape Breton Nova Scotia II. Pyrite and its alteration products. Canadian Mineralogist Vol. 17, pp 63 - 70.

APPENDIX 1

Bacterial Media and Nutrient Solution

1. The growth medium used for culturing of Thiobacillus ferrooxidans is the modified Tuovinen and Kelly Medium (Table A).

2. The nutrient solution used in the actual leaching process for recirculation of the organisms consists of the above medium, without the iron source, ferrous sulphate, added.

3. To check for viable cultures an aliquot of 100mls of the sample is mixed with 900mls of Tuovinen and Kelly medium. If after several days to 1 week the solution turns deep red, then there is growth in the original sample. If there is no change after 10 days then it is assumed that there is no viable culture.

TABLE A

Modified Tuovinen and Kelly Medium

1. 0.4 g ammonium nitrate
   50.0 mg potassium phosphate dibasic
   50.0 mg magnesium sulphate
   -add to 900 ml distilled water.

2. 33.3 g ferrous sulphate (filter sterilized)
   -add to 900 ml of previous solution.
   -adjust pH = 1.8 with sulphuric acid.

1. Modified from medium described in R.G.L. McCready (1984) microbiological studies on high sulfur coals, pg. 65.

APPENDIX 2

Fe3+ Determination

The determination of the ferric ion content of the leaching solution is based on the spectrophotometric absorption of light of a specific wavelength by an acidified solution of sample.

1. A standard curve was prepared on a Spectronic 20 spectrophotometer at the laboratories of the University College of Cape Breton in the following manner:

   a. Prepare standard 100 ppm (Fe^{3+}) solution by addition of 4840mg of $FeCl \cdot 6H_2O$ in 1000 mls of distilled water.

   b. Prepare dilution series as follows:

100 ppm solution (ml)	H_2O (ml)	HCl(2N) (ml)	CONC. (ppm)
0.2	1.8	2.0	5
0.4	1.6	2.0	10
0.6	1.4	2.0	15
0.8	1.2	2.0	20
1.0	1.0	2.0	25
0.0	2.0	2.0	blank

c. Analyse an aliquot of the blank to zero the instrument at 410 nm. Measure the absorbances of each dilution and plot data to obtain standard curve.

2. To analyse a sample, zero the instrument and prepare the sample as follows. 2 parts sample: 18 parts H_2O : 20 parts HCl(2N). This represents a 20:1 dilution (2 in 40). Measure the absorbance of a portion of this sample at 410 nm as before. If the absorbance measured is outside the range of that covered by the standard curve, adjust the dilution ratio accordingly.

NOTE: If the sample contains fines, the analysis can be affected. If this is the case the sample is filtered using a 0.45 um Millipore cellulose ester filter.

3. Read the corresponding concentration for the measured absortion from the standard curve and adjust the figure to reflect the dilution to obtain the Fe^{3+} concentration in the sample.

APPENDIX 3
SEM-EDX SAMPLES - PHOTOGRAPHS

CONTROL PILE:

1. 85-185-03
   Sample exhibiting whitish crust on coal surface.

2. 85-185-05
   Sample exhibiting orange-yellow globular material.

INNOCULATED PILE:

3. 85-185-14
   Sample exhibiting both whitish and yellow globular material.

4. 85-185-17
   Sample exhibiting single clumps of orange-yellow material

5. 85-185-18
   Sample exhibiting distinctly orange crusted material.

APPENDIX 4
SEM PHOTOMICROGRAPHS OF HYDRATED SULPHATES

SAMPLE 85-185-17

PHOTO #

1. 102104, 75 X, Secondary Electron Image (SEI) low magnification image of blooms of disk-like hexagonal crystals (see 5) and fibrous material.

2. 102103, 350 X, SEI
Enlargement from 1 showing fibrous material

3. 101003, 750 X, SEI
Medium magnification image of fibres and crystal blooms as in 1.

4. 100801, 1000 X, SEI
High magnification image of crystal blooms as in 1.

5. 101002, 2000 X, SEI
High magnification image showing stacked arrangement and hexagonal shape of crystals as in 4.

6. 102101, 3500 X, SEI
EDX analysis of crystal (normal to beam axis) at crosshairs gives Fe, S peaks (spectrum 1) typical of spectra acquired for most crystals.

7. 102102, 3500 X, SEI
EDX analysis of fibrous material at crosshairs gives Fe, S, Al peaks (Spectrum 2)

SAMPLE 85-185-05

8. 111201, 1500 X, SEI
High magnification image of stacked, hexagonal, disk-like crystals EDX Spectrum gives Fe, S peaks with minor Si, Mg (spectrum 3).

SAMPLE 85-185-14

9. 123106, 100 X, SEI
Low magnification image of fibrous material associated with nodular crystal growths (see 11 & 12).

10. 123107, 350 X, SEI
Enlargement of fibrous - crystalline interface area from 9.

11. 123103, 350 X SEI
Medium magnification image of nodular crystal growth.

12. 123105, 500 X, SEI
EDX trace at crosshairs of nodular crystal gives Fe, S peaks with minor Si Mg (Spectrum 5).

13. 123108, 2000 X, SEI
Close-up of fibrous growth, EDX spectrum gives Fe, S, Al peaks with minor Si (Spectrum 4).

14. 123111, 350X, SEI
Medium magnification image of blooms of crystals with surrounding and intermixed pyrite framboids (see 15).

15. 123110, 350 X, Backscattered Electron Image (BEI)
Backscatter Image of 14 showing pyrite crystals and framboids (bright), Fe, S, Al crystal blooms (grey) on coal surface (dark).

16. 123109, 200 X, SEI
Close-up of Fe, S, Al crystal blooms (Spectrum 6) and pyrite crystal clusters and framboids

SAMPLE 85-185-03

17. 123001, 50 X, BEI
Low magnification image of pyrite crystal clusters and framboids (bright); Fe, S, Si, Al material (grey) on coal surface (dark).

18. 123102, 50 X, SEI
Secondary Electron image of 17.

19. 123004, 200 X, BEI
Enlargement of upper right corner of 17 showing pyrite crystals and framboids (EDX 2, Spectrum 8) and Fe, S, Si, Al material (EDX 1, Spectrum 7).

SAMPLE 85-185-18

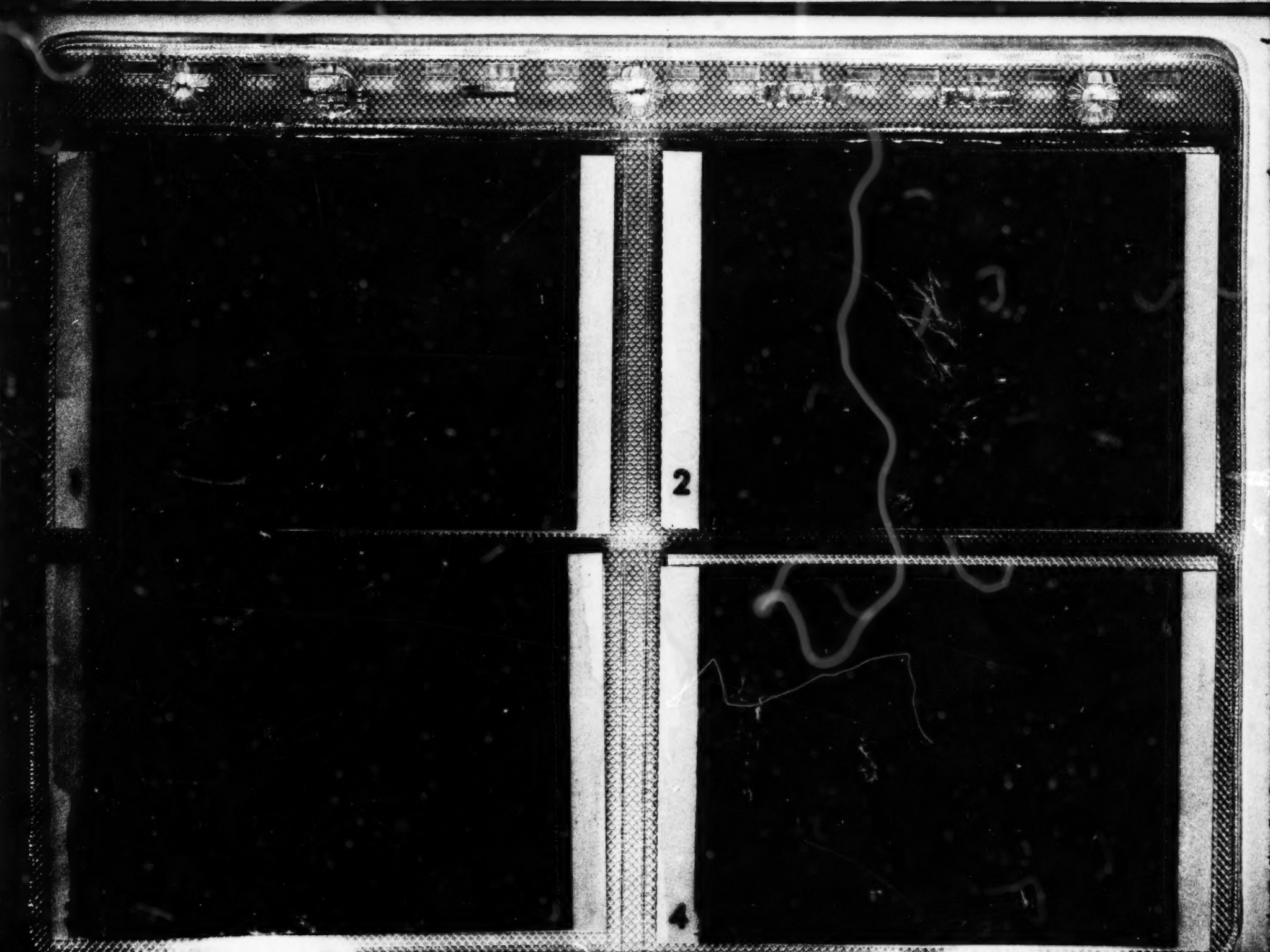
20. 123118, 35 X, SEI
Low magnification image of crystal blooms.

21. 123120, 350 X, SEI
Enlargement of crystal blooms from 20.

22. 123121, 3500 X, SEI
Close-up of crystals from 21. EDX spectrum gives Fe, S peaks with minor Si (may be artifact) (Spectrum 9).

23. 111301, 350 X, SEI
Medium magnification image of crystal clusters with fibrous material (see 24).

24. 111302, 3500 X, SEI
Close-up of centre portion of 23, EDX spectrum of fibre "root" gives Fe, S, Al, Si peaks (Spectrum 10).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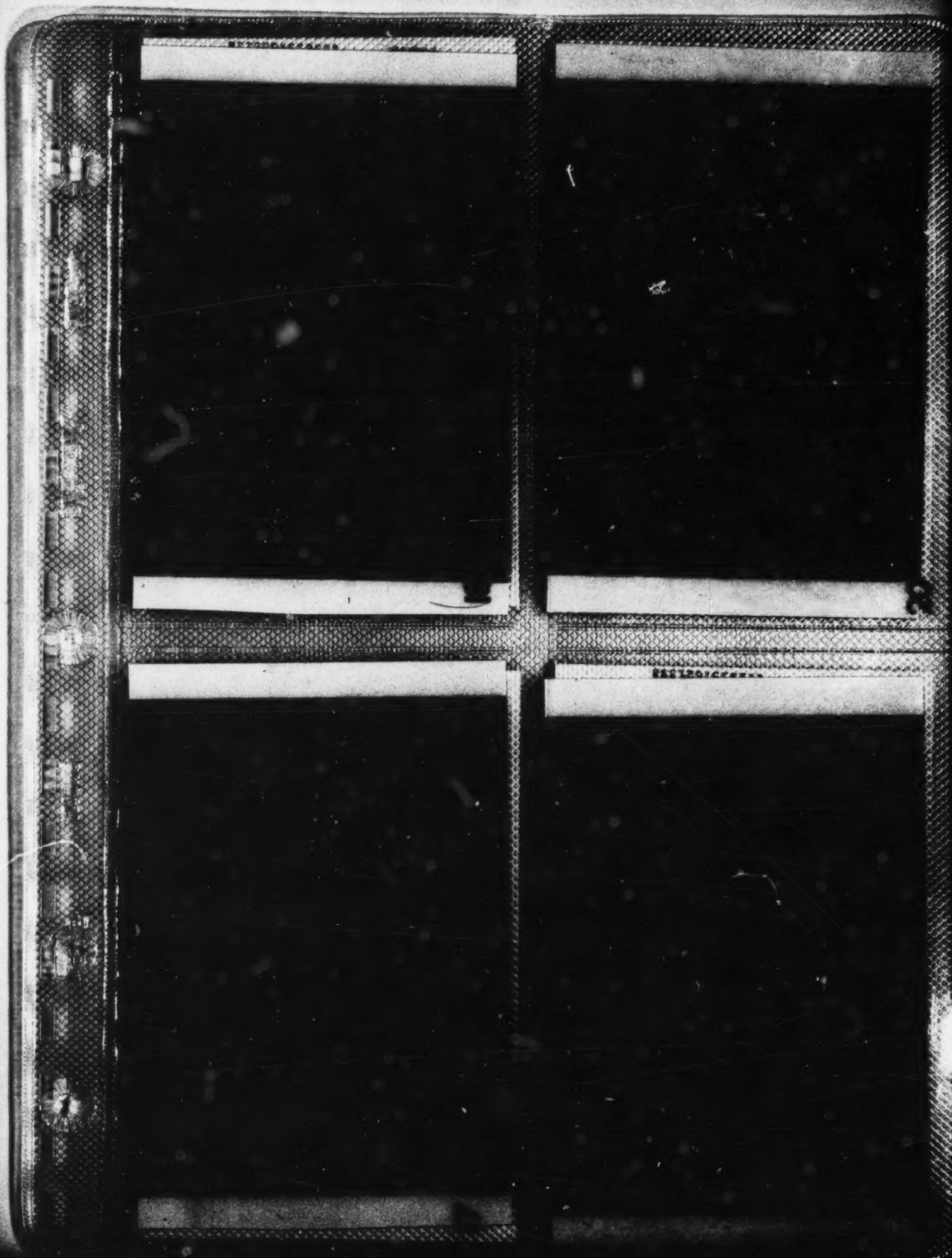
9

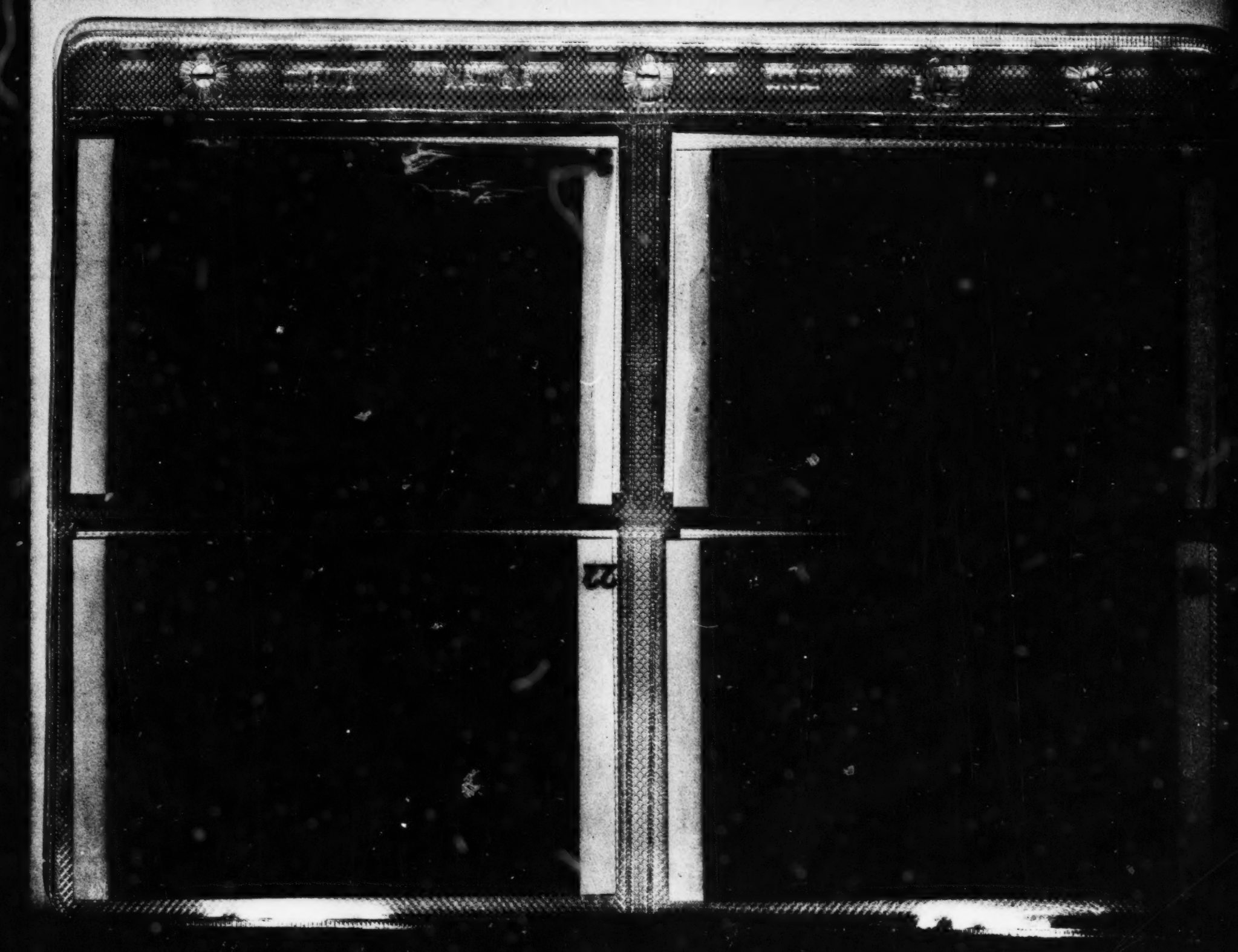
8

10
12

14

16





APPENDIX 5

ATLANTIC COAL INSTITUTE

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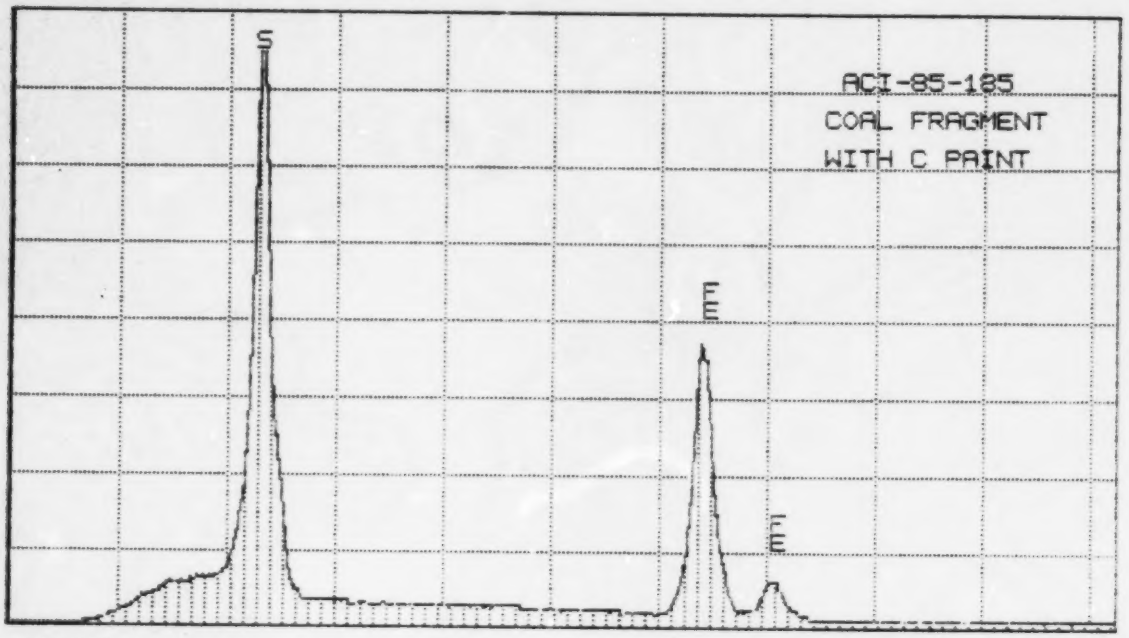

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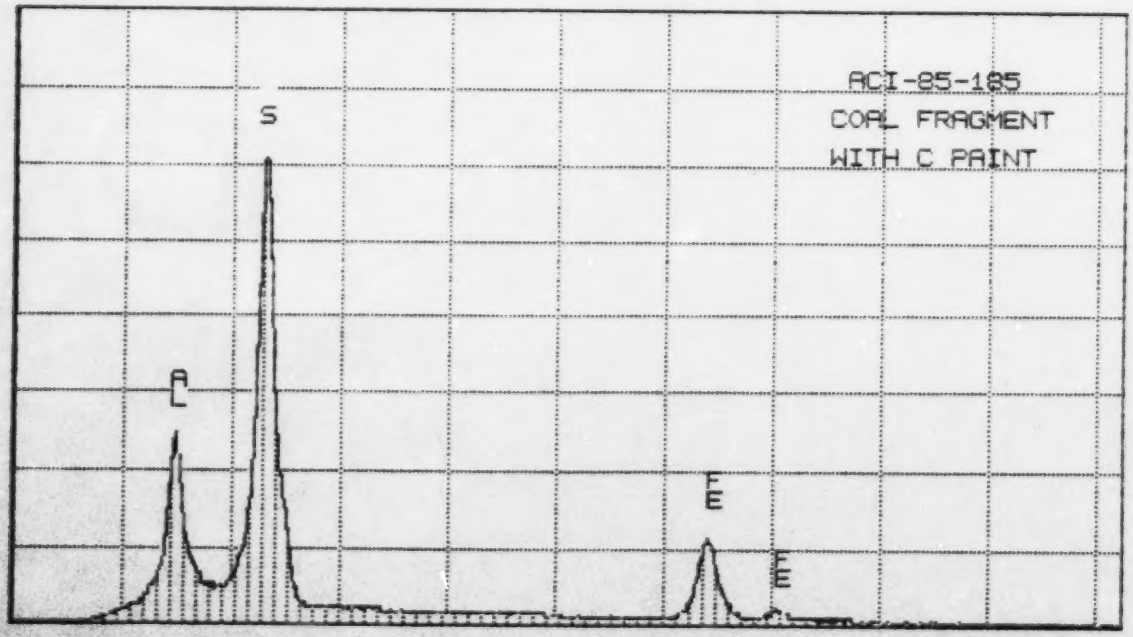

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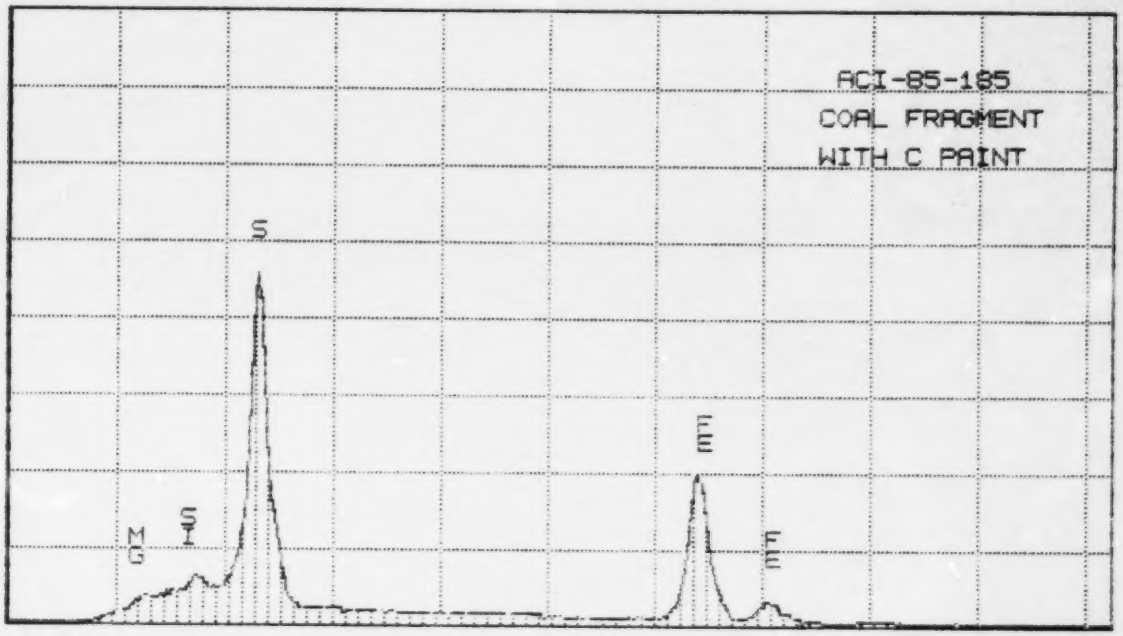

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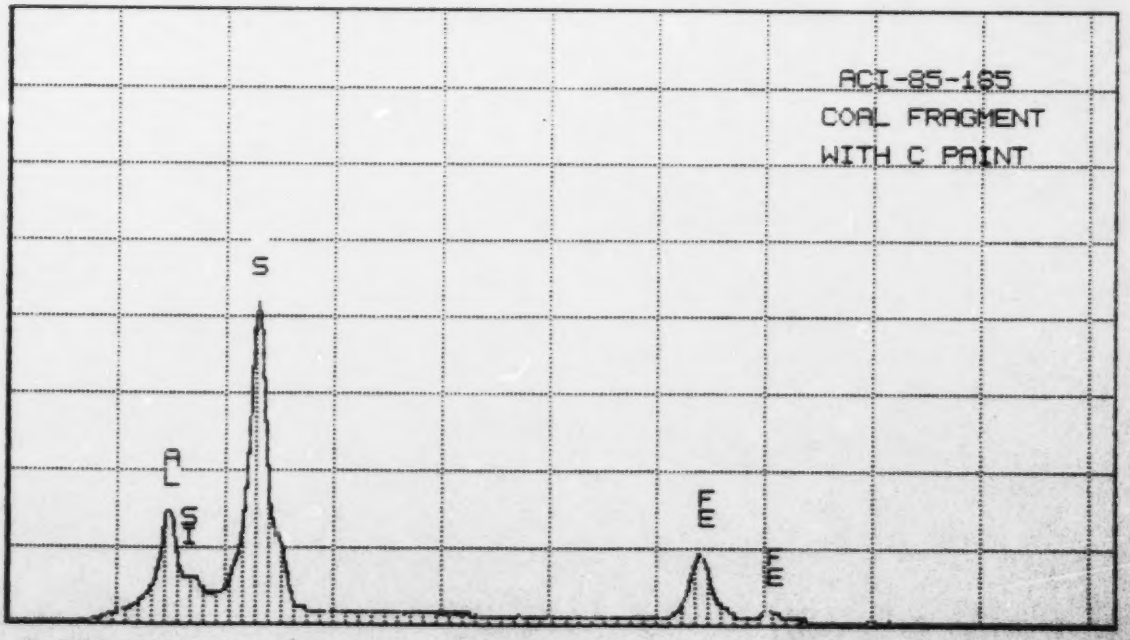

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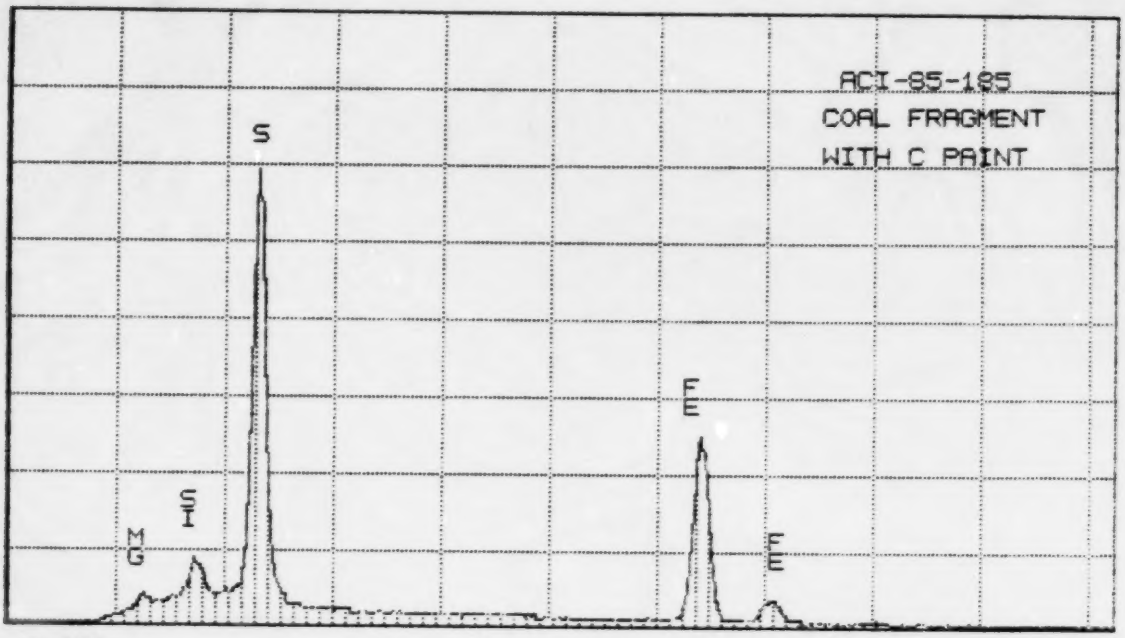

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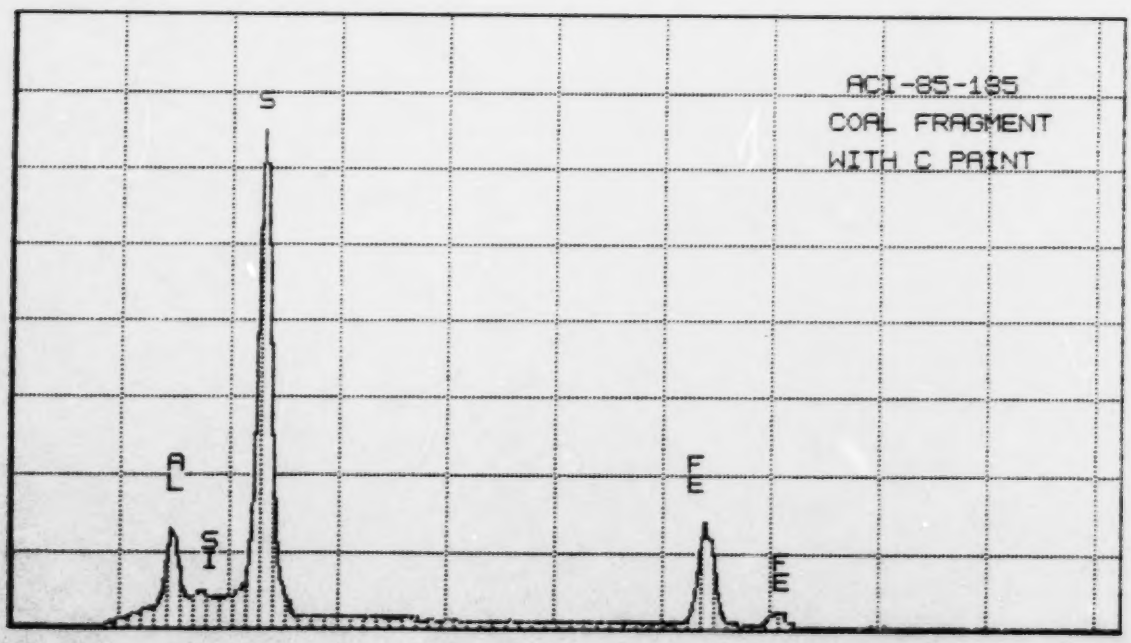

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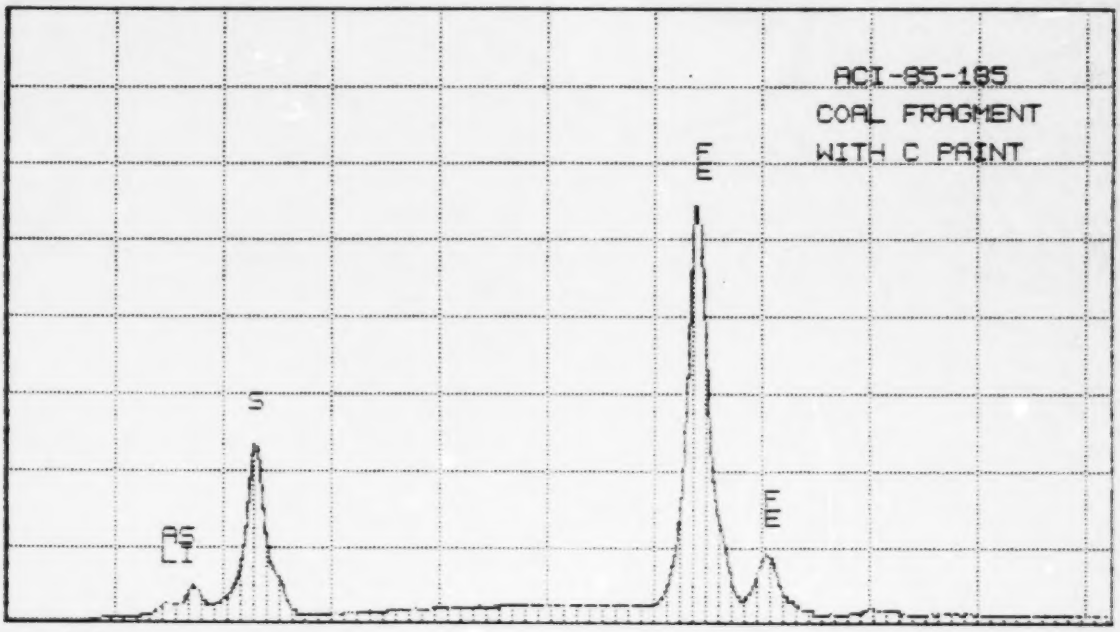

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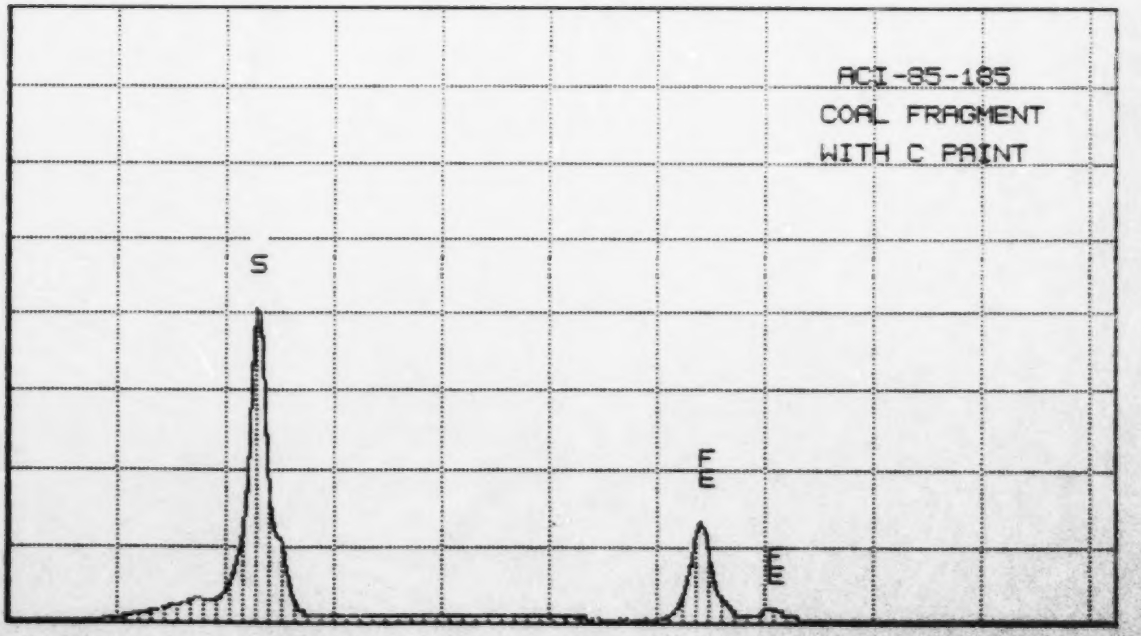

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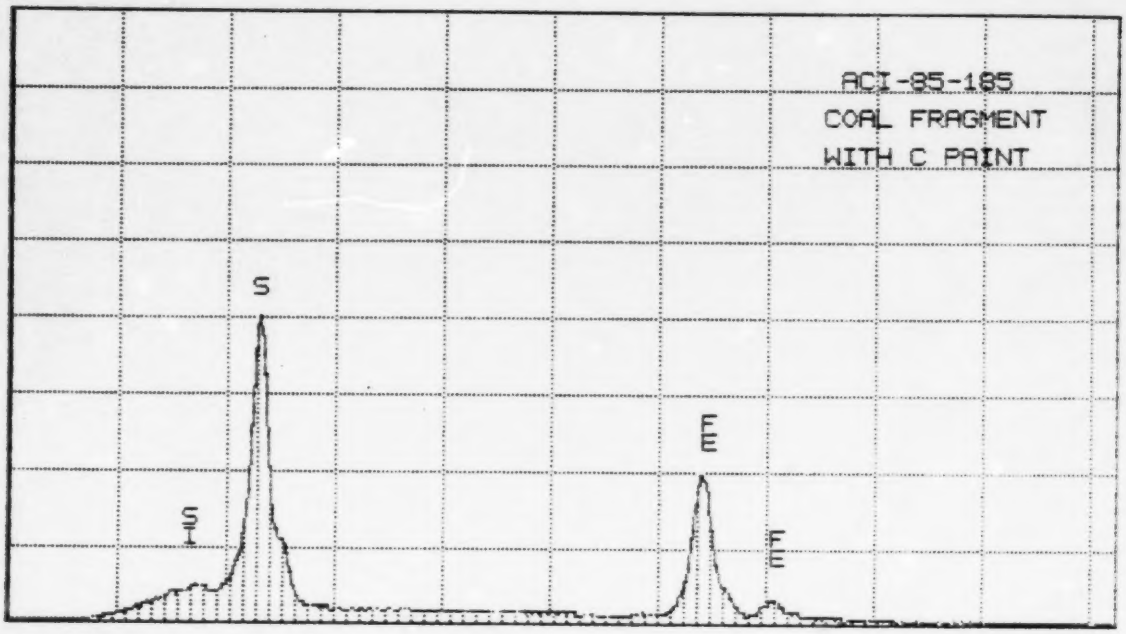

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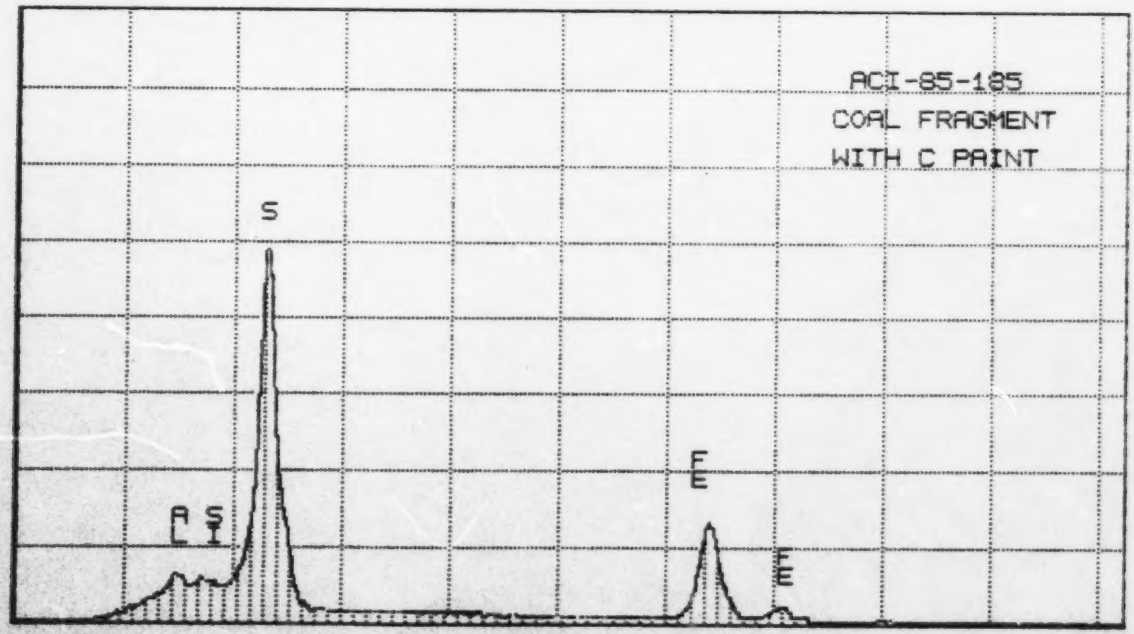

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10

APPENDIX 6

LEACHATE SAMPLES

DATE SAMPLED	SAMPLE	Zn ppm	Co ppm	Ni ppm	Mn ppm	Al ppm	Sr ppm
NOV. 11/85	CONTROL	32	4.50	10	92	425	0.16
NOV. 11/85	INNOCULATED	25	3.06	6.50	66	288	--
DEC. 11/85	CONTROL	0.03	0.06	0.05	--	--	--
DEC. 11/85	INNOCULATED	0.1	0.09	0.18	0.78	--	--
MAR. 17/86	CONTROL	72	4.41	12	34	140	1.00
MAR. 17/86	INNOCULATED	73	4.07	10.8	36	163	0.90

LEACHATE SAMPLE
INNOCULATED - MARCH 17,1986

Ag	ppm	<90	Li	ppm	0.55
As	ppm	82	Lu	ppb	6.20
Au	ppt	200	Mn	ppm	40.1
B	ppm	<0.5	Mo	ppb	2100
Ba	ppb	<10	Na	ppm	349
Be	ppm	<0.1	Nd	ppb	120
Bi	ppm	0.13	Ni	ppm	12
Br	ppb	<20	Pb	ppm	1.3
Ca	ppm	<300	Rb	ppm	<10
Cd	ppm	0.10	Sb	ppb	14
Ce	ppb	127	Sc	ppb	90.0
Co	ppb	4600	Se	ppb	910
Cr	ppb	490	Sm	ppb	40.0
Cs	ppb	<20.0	Ta	ppb	<8.0
Cu	ppm	6.6	Tb	ppb	8.0
Eu	ppb	5.40	Th	ppb	86
Fe	ppm	17100	U	ppb	2.5
Ge	ppm	<0.3	V	ppb	<2
Hf	ppb	<6.0	W	ppb	<4
Hg	ppb	120	Yb	ppb	40.0
K	ppm	<40	Zn	ppm	41
La	ppb	98.0			